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Treatment of lead and zinc slags in hollow electrode DC-EAF in consideration of calculated phase equilibria and thermodynamics

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The legislation for the disposal of heavy metals contaminated substances is still going to be tightened up in the future. The main criteria for the utilization or deposition of these residues are the content and the water solubility of these metals. Through this, the use of slags from the lead- and zinc-industry in road building may become limited or even not possible at all. From an economic point of view this would lead to Pb/Zn losses and costs for safe dumping.

This paper investigates the feeding of the reducing agent via hollow electrode DC smelting and the yield of metal reduction. After carrying out thermodynamic calculations, the process window conditions for electric-arc furnace tests were determined. Doing this, the zinc could be reduced, evaporated and recovered as an oxidic intermediate product flue dust, while the lead was present either in flue dust or in metallic form. The zinc content in the final slag could be reduced by up to 90%. The slag product has undergone an eluation test which allows it to be used as building materials.

Keywords: zinc reduction, lead reduction, hollow electrode, slag treatment, electric arc furnace

Introduction

Zinc and lead containing residues, such as slag from the lead or zinc industry, leaching residues or flue dusts, often contain pollutants like heavy metals or anions[12]. Dumping of residual substances like metallurgical slags is made more difficult by stricter requirements for environmental protection and steadily rising costs worldwide. Besides, since the introduction of the Basel Convention for controlling the international transport of environmentally hazardous residual substances (1989), reprocessing of residual substances has become of special interest[3]. The main criteria for deciding whether to use or dump these residual substances are the content of heavy metals and their water solubility. Thus the use of slags from zinc or lead industries as construction material may become limited or even forbidden. Furthermore, the high zinc and lead contents in the used or deposed slag constitute metal losses, which are unfavourable from the economic point of view.

In a three-year collaborative project with RWTH Aachen University and industry partners Sudamin MHD, Berzelius Stolberg and Varta Recycling, which is funded by the Federal Ministry of Education and Research, a process has been developed to reach zinc and lead values according to future expected regulations using a subsequent slag treatment in an electric arc furnace, by hollow electrode technique.

This paper will show calculations of the thermodynamic and kinetic boundary conditions for this process. Furthermore, it is shown in test runs that a slag product suitable for building materials, a zinc-rich flue dust and a metallic lead phase can be produced by this technology.

Oxidic slags from thermal zinc and lead production

For worldwide primary and secondary lead winning, several processes are in use, i.e. the lead shaft furnace process, QLS process, KIVCET process or VARTA shaft furnace. Because of the low solubility of lead in aqueous solutions, hydrometallurgical winning cannot be applied economically, so that primary lead production takes place in the pyrometallurgical way without exception. Most of the lead winning processes lead to an oxidic slag system.

Contrary to lead winning, zinc primary and secondary winning usually takes place typically by leaching and electrowinning. The only pyrometallurgical procedure for zinc winning that plays a substantial role nowadays is the Imperial Smelting process, which also serves for lead production[10]. In the past, only primary raw material was treated, but meanwhile the aggregate was also used intensively for the processing of residual substances. The products of an IS furnace are zinc, crude lead and a oxidic slag that collects the iron as olivine. Nevertheless a significant sulphur concentration—as sphalerite and wurtzite—is measurable in the oxidic slag.

In the IS as well as the QSL and KIVCET processes slag is granulated in a water spray. The high Zn content of the slag represents a zinc loss that cannot be avoided because a further Zn reduction would be accompanied by the increased risk of an iron reduction, which leads to formation of solid iron in the shaft and to damage of the refractory lining. An increase of the process temperature in order to keep the iron liquid would result in intensified energy consumption, an additional liquid phase (e.g. pig

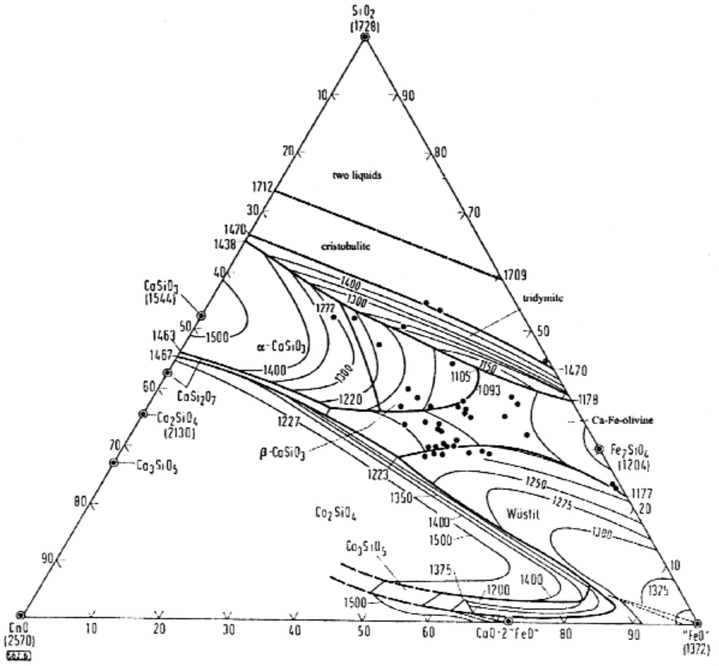

Figure 1. Phase diagram CaO-FeO-SiO_2 of zinc and lead slag systems[19]

iron) and increased lead evaporation. Most of the lead content in the slag is bounded physically as drops of crude lead.

A low liquidus temperature as well as a low viscosity and a minimization of metal losses are the most important characteristics of the slags. The liquidus temperatures of some oxidic lead and zinc slags (referred to as the system FeO-CaO-SiO_2) are within the range of approx. 1000°C–1200°C as shown in Figure 1.

Developments for the treatment of lead and zinc slags

Slag fuming

Slag fuming is an evaporation process in which zinc and lead from a liquid slag are transferred into an oxidic flue dust[13]. The liquid slag is charged into the water-cooled reactor, where also solid residues, such as slag granulates can be charged[10]. Pulverized coal (<0.1 mm) is injected into the slag bath by air[6]. At the beginning of the process, as well as before tapping, the air/coal ratio is increased briefly in order to increase the bath temperature by coal combustion. The metal oxide reduction takes place at 1150°C–1300°C according to the following equation:

$$ZnO(1) + CO(g) = Zn(g) + CO_2(g) \qquad [1]$$

Above the slag bath the metal vapour is oxidized with tertiary air, and the offgas is post combusted:

$$Zn(g) + \frac{1}{2}O_2(g) = ZnO(s) \qquad [2]$$

$$Co(g) + \frac{1}{2}O_2(g) = CO_2(g) \qquad [3]$$

Subsequently, the gas is cooled and the zinc containing dust is collected in filters and/or dust precipitators. The use of the offgas heat (e.g. for power generation, preheating of injected air) is decisive for the economical efficiency of the process[6]. Slag fuming is generally a batch process[15], usual reduction times vary between 50 and 100 minutes, but they can reach up to three hours[17,18]. The final Zn content in the slag typically amounts to 1.5%–3.3%, but values below 1% can be reached with very long reduction times[4,18]. The lead content at the end of the slag fuming amounts to 0.03%–0.05%[6].

Disadvantages of slag fuming are the large quantities of exhaust gas and the complexity of the offgas treatment. The operation of a slag fuming plant is only feasible if an appropriate steam consumption is given[10]. Another disadvantage is the high demand for coal due to its double application as fuel and reducing agent. Besides, it is impossible to recover zinc in metallic form because of the high oxygen potential in the exhaust gas, so that the produced zinc oxide has to be reduced again in a subsequent step. The product of the slag fuming process is a mix oxide, which bears most of the lead-input as PbO or PbS. To avoid interferences in the subsequent zinc winning process, a clinker process has to follow.

Ausmelt or Isasmelt process

A more recent process suitable for the removal of lead and zinc from slags is the Ausmelt or Isasmelt process. As the case of slag fuming, coal as reducing agent and solid fuel is injected into the molten slag. But instead of using tuyeres for this purpose, as in slag fuming, the injection is done through a lance[14]. The final slag contains <0.1% lead and <1.0% zinc and can be used e.g. for road construction[11]. This process differs from the slag fuming procedure only in equipment design and thus bears similar disadvantages: large quantities of exhaust gas, a complex offgas system, a high demand for coal, and a reclamation of a mixed-oxide.

Computational modelling of thermodynamic data for reaction equilibra in oxidic slag systems

Thermodynamics makes it possible to gain knowledge about the reachable equilibrium in the process, as well as to obtain the conditions that are suitable to recover zinc and lead from their slags. Only if the characteristics of the components, in particular the activity, are known are exact statements possible.

For this purpose the activity coefficient and/or activity of oxides in liquid slag systems is modelled. Models from literature as well as a computer database are used. The models presented in literature provide a basis for the computation of the oxygen partial pressure. For the calculations and data comparisons presented in this paper models of Battle and Hager, Kellog, Conradt and Jak and Hayes have been selected[2,5,8,9].

Computer models that are based on databases allow one to evaluate the properties of the slag more exactly. Hence it can be concluded at which temperatures zinc and lead can be reduced and evaporated if necessary, how large the required amount of carbon is, and how to inhibit the iron reduction during slag treatment. Due to the progress of past years in the domain of data processing technologies it is now possible to use PC technology for the computer-based modelling of problems such as the treatment of slag.

The software FACTSage was developed in 2001 by the unification of the programs FACT Win and ChemSage[1]. The basis for the computations in FACTSage are databases in which the relevant values for pure materials and solutions are stored.

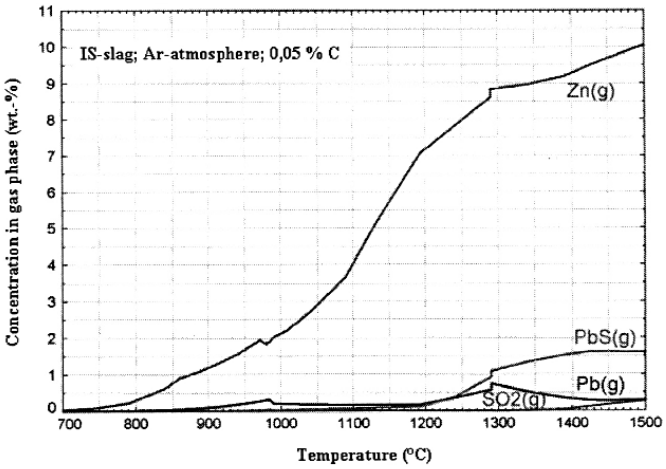

Figure 2. Modelled composition of gas phase in IS-slag treatment with an addition of 0.5% carbon

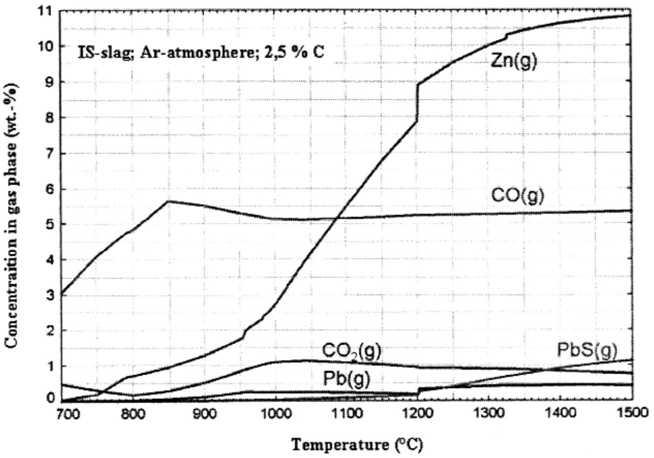

Figure 3. Modelled composition of gas phase in IS-slag treatment with an addition of 2.5% carbon

Within the scope of this work FACTSage was used in order to model the equilibria of the examined slags in a temperature range of 1000ºC–1500ºC. Temperature, carbon addition and gas phase were given as process conditions in order to obtain data for the zinc and lead yield. The specification of a gas phase—in this case as a neutral gas phase—is necessary in order to permit the evaporation of components. If the existence of a gas phase is not given, FACTSage cannot compute an equilibrium between solid, liquid and gas phase. The basis of the modelling was the chemical analysis of the slags and the database FACT in the version 5.00. X-ray analyses help to select the relevant phases for FACTSage calculations.

On this basis, the amount of zinc and lead in gas phase under different conditions has been calculated. Figures 2 and 3, in which the resulted gas phase composition is shown for the equilibrium, allow an evaluation of the possibilities of the zinc and lead output in the gas phase. Because of the reducing conditions caused by the addition of carbon, the majority of zinc will be vaporized. The lead amount in gas phase decreases with higher temperatures and higher reducing conditions.

It has to be noted that in the arc furnace process the output of lead as an oxidic flue dust should be avoided. According to other studies it has to be presumed that 80% of the lead contained in the slag is present in metallic form[7]. It has to be investigated to what extent lead forms a separate metal phase that does not reach the equilibrium. Due to the small offgas volume of the arc furnace process and the avoidance of lead oxidation, the quantity of lead as a separate metal phase will be higher than in slag fuming or the Ausmelt process.

Thermodynamics and kinetics of zinc reduction

Thermodynamics of zinc reduction

The following equation generally applies to the reaction between zinc and oxygen:

$$2Zn + O_2 = 2ZnO \quad with \quad K = \frac{a_{ZnO}^2}{a_{Zn}^2 \cdot p_{0_2}} \qquad [4]$$

The oxygen potential at the thermodynamic equilibrium is represented by the following equation:

$$RT1np_{0_2}\Delta H^\circ - T\left(\Delta S^\circ - R1n\frac{a^2{}_{ZnO}}{a^2{}_{Zn}}\right) \qquad [5]$$

with ΔH^o: standard enthalpy or reaction (J/mol); ΔS^o: standard entropy of reaction (J/mol K); T: temperature (K); R: 8.314 J/K; K: equilibrium constant; a_i: activity of the element i; p_{O2} : O_2 partial pressure (Pa)

For a rough calculation the activities are often presumed to be approximately one, but as this approach neglects the influence of the activities, it does not mirror the real conditions. The activity is linked with the mole fraction by a coefficient:

$$a_i = \gamma_i x_i \qquad [6]$$

with a_i: activity of the element i; γ_i: coefficient of activity of the element i; x_i:mole fraction of the element i

Previous studies investigated different values for the activity coefficients in slags from lead and zinc industries [2, 5, 7, 8, 9]. A new FACT calculation gives different values for a_{ZnO}. Figure 4 shows that the different models do not exert significant influence on the calculation of the zinc activity in the liquid slags investigated in this work. The data are based on chemical analyses of an ordinary QSL slag.

Kinetics of zinc reduction

The kinetics of zinc reduction were particularly examined for the slag fuming process by various authors[13, 15, 20]. About the reaction mechanism and the speed-determining reaction step contradictory theories prevail, which are summarized here.

According to Bell, Turner and Peters the large surface and the strong bath circulation during slag fuming lead to a very fast mass and heat exchange, so that the thermodynamic equilibrium (Equation 4) between slag and gas is always reached[13, 18]. The zinc evaporation is directly related to the carbon supply, a constant addition of coal causes the zinc content in the slag to decrease linearly[9, 15]. According to Quarm, the zinc reduction is a first order reaction concerning the ZnO content[15]. The reaction rate is shown by the following equation:

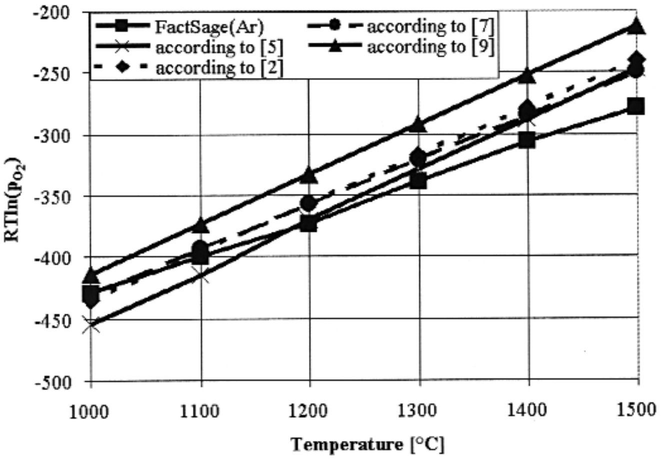

Figure 4. Oxygen potentials of ZnO in QSL slag as functions of the temperature

$$\frac{d}{dt}c_{ZnO} = -k_1 c_{ZnO}(t) \qquad [7]$$

with k_1: reaction rate constant (min^{-1}); c_{ZnO} (t): ZnO concentration in the slag at time t (wt.-%); *t*: time (min)

By integration of this equation the degression of the ZnO concentration can be computed:

$$c_{ZnO}(t) = c_{ZnO;t=0} \bullet \exp(-k_1 \cdot t) \qquad [8]$$

However, a linear reduction of the ZnO content was observed in the production process.

The zinc reduction rate is primarily determined by the Boudouard reaction, so that the formation of a carbon monoxide bubble around an injected coke particle is a substantial procedure. Subsequently, the material transfer in the slag becomes the speed-determining step. The zinc gas pressure becomes more and more crucial at higher zinc concentrations. A turbulent circulation of the slag and a high temperature accelerate this effect[7].

For zinc reduction by slag treatment, a high bath turbulence is essential. This is realized by an intensive gas flow in the slag fuming process. Electric arc furnaces realize the requested extreme mass transfer[16].

Zinc and lead reduction tests in a pilot scale DC arc furnace by hollow electrode system

Figure 5 presents the pilot scale arc furnace at IME Aachen with an operational power rating of approximately 0.5 MWh. The electric current supply of the furnace is secured by a 0.5 MVA electric rectifier. The furnace is provided with a water-cooled bottom anode of copper and a hollow graphite electrode. The cathode's vertical position is adjustable by an electrical system. The hollow electrode system for charging lignite coke into the slag has been developed at IME. The system is gastight: only a low gas flow of nitrogen is needed to counteract the capillary forces between slag and the inner electrode surface. After the reaction, the liquid phase is tapped by the bottom tap hole. An exhaust gas system collects the offgas and leads it to a water-cooled spark separator and a bag filter, where the flue dust is separated.

The inlet and outlet temperatures of the cooling water, various temperatures of the furnace lining and temperatures on different positions of the bottom electrode and the offgas system are continuously monitored. The melt's temperature is measured discontinuously by a thermocouple lance.

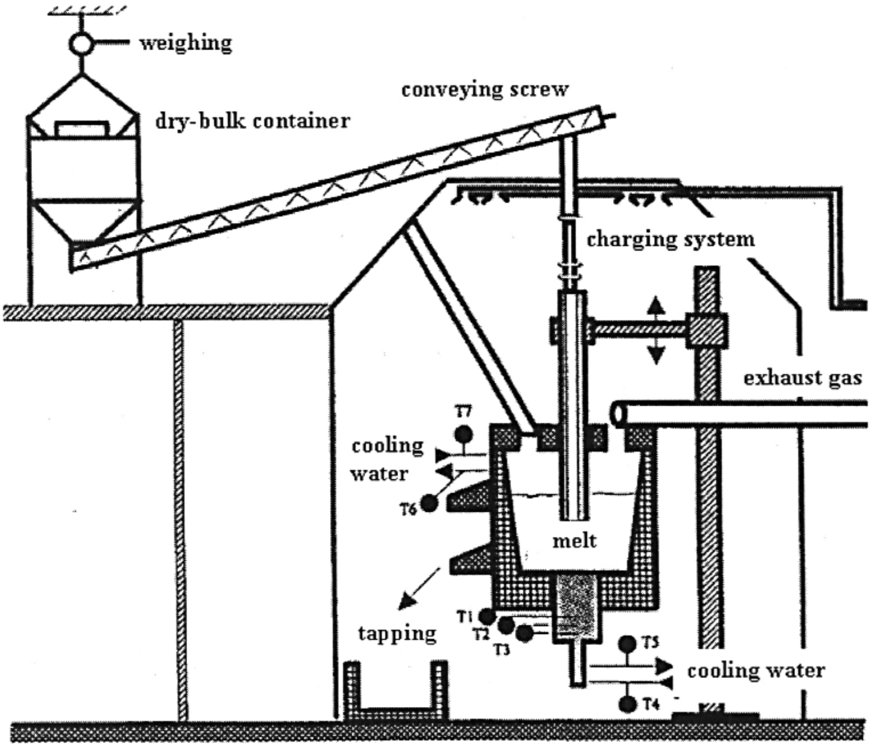

Figure 5. Schematic structure of the IME-pilot arc furnace; T_i: thermocouples

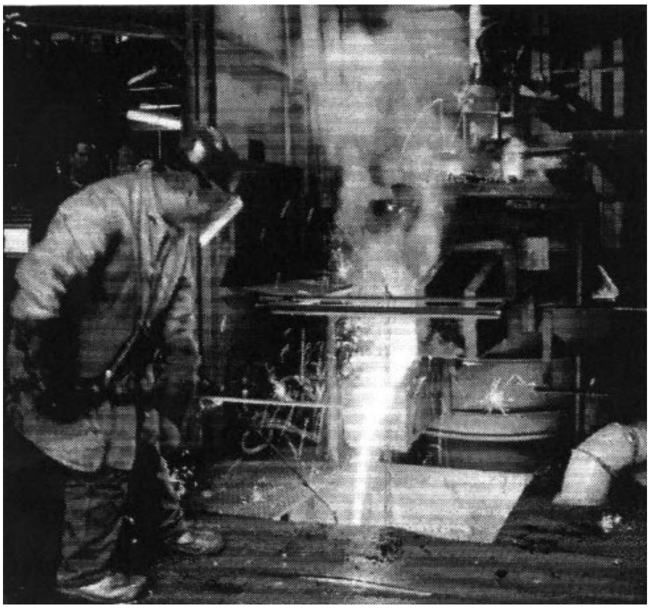

Figure 6. Tapping of the treated slag

In preliminary lab scale-tests (10 kg scale) the melting and reduction behaviour of the slag are examined. Slag from the QSL-process with a content of 12.7% zinc and 9.6 % lead has been treated for this investigation. The results mirror that tapping at 1500°C is successful due to low grades of slag's viscosity. Therefore approx. 1500°C is aimed at as the tapping temperature for the pilot test run series.

At the beginning of each test run 250–300 kg of solid slag granulate is charged through the furnace top and melted. Charging of liquid material to simulate a production process is not possible at IME because there is no equipment for keeping this quantity of slag in the liquid state. Therefore the analysis of the solid IS slag (5.06% Zn; 1.09% Pb; 25.5% Fe) is not considered as the initial value. After a smelting time of about 45 min, a slag sample is taken. Its composition serves as the start value (2.58% Zn; 0.48% Pb; 31.1% Fe). When the major part of the charged slag is molten, coke is charged through the hollow electrode for about one hour. During that time most of the reduction takes place. A reduction and settling time of about one hour follows.

During the process slag samples are taken from the furnace. After tapping and solidification of the slag, the slag block is removed, crushed, milled and sampled to be analysed. At the same time samples of the flue dust are taken from the filter system.

Results of zinc reduction in IS slag

Figure 7 shows the progression of the zinc concentration curve during a reduction experiment as a function of time.

During the first 20 minutes, the zinc concentration rises. This effect can be explained by the fact that the sampling always takes place close to the electrode. In this zone, slag melting and zinc reduction by electrode's graphite start immediately and zinc is evaporated. When the slag in the external area becomes molten, it mixes with the slag from the central area, so that the zinc content becomes more homogeneous after the complete melting of charged slag. During this time zinc reduction is controlled more or less by the thermodynamic equilibrium.

Between 20 and 70 minutes of reduction time, an almost linear reduction of the zinc content can be observed, which coincides with the theory mentioned above. Then the zinc reduction is slowed down. Between the last sampling at t = 68 min and tapping at t = 135, min the zinc content decreases from 0.3% to 0.1%. Thermodynamically, the lower Zn-reduction ratio at the end of the experiment can be traced back to the reduced ZnO activity. From the kinetic point of view, the slowing down of the zinc reduction can be explained by the less favourable mass transfer of the very low concentrated Zn^{2+}-ions to the reaction place. Both effects will superimpose each other. This figure shows that a zinc content in slag lower than 1% can be achieved after a reduction time of less than one hour in an arc furnace using carbon insertion via the hollow electrode.

With the IME equipment it is not possible to lead the flue gas steam to a condenser, in which it would be possible to separate the zinc in metallic form. This has to be done in a pilot plant. So a re-oxidized flue dust with a content of 81 % ZnO and 19 % PbO is separated. Tables I and II show

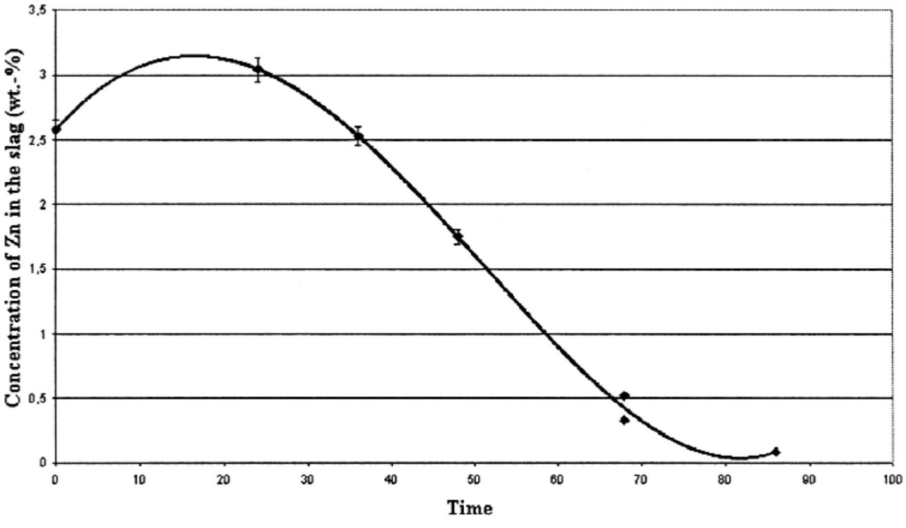

Figure 7. Zinc content in IS slag as a function of time

Table I
Analyses of charged and treated slag and resulted flue dust of QSL-slag

QSL-slag	Pb	Zn	Fe_{total}	Fe^{2+}	SiO_2	CaO	Al_2O_3	MgO	S
Before treatment	8.92	11.70	28.20	18.10	25.10	9.86	2.30	1.26	0.10
After treatment	0.11	0.72	41.50	32.80	n/a	n/a	n/a	n/a	0.095
Flue dust	41.70	44.20	<0.10	<0.10	n/a	n/a	n/a	n/a	n/a

Table II
Analyses of charged and treated slag and resulting flue dust of IS-slag

IS-slag	Pb	Zn	Fe_{total}	Fe^{2+}	SiO_2	CaO	Al_2O_3	MgO	S
Before treatment	1.10	11.20	25.50	22.60	21.60	16.40	7.86	4.39	2.45
After treatment	0.02	0.16	29.00	24.80	n/a	n/a	n/a	n/a	1.90
Flue dust	21.50	61.40	0.40	0.40	n/a	n/a	n/a	n/a	n/a

the analyses of the charged QSL- and IS-slag and the results after slag treatment by hollow electrode system, in EAF.

Results of lead reduction in IS-slag

Since approx. 80% of the lead input are bound as metal, the settling effect becomes more important. In contrast to the reduction of zinc, the lead concentration follows a non-linear function earlier (after approx. 40 min). In order to reduce the lead concentration in slag to less than 0.1% a reduction time of more than 70 minutes is needed (Figure 8).

Results of iron reduction in IS-slag

Figure 9 shows the distribution of different oxidation stages of iron in the examined IS slag. In the charged slag the main part of iron is bound as FeO (i.e. as Fe^{2+}), only a small part is bounded as $FeO•Fe_2O_3$ (i.e. Fe^{3+}) or metallic iron. During the slag treatment in EAF more Fe^{2+} is reduced to Fe^0. Due to the very high local temperatures—achieved by the arc—and the good mass flow in the liquid slag, no solid pig iron will be present in the furnace. It is assumed that a part of this iron serves as a reduction agent for the zinc reduction. These results are confirmed by FACTSage 5.2 calculations, in which the iron is assumed as ideally solved in the slag.

Summary

The development of a new technology for slag treatment is pushed by expected restrictive legislation that demands low heavy metal contents in residual substances. The charging of coke and absence of intensive gas insertion avoid high offgas volumes. The temperature that is necessary for the process can be adjusted without any problems in the electric arc furnace. A reduction of iron can only partly be inhibited; but local formation of metallic iron is not problematic since solidified iron will easily be remelted by the electric arc.

The investigations point out that the combination of arc furnace processing and charging of carbon bearing material by hollow electrode technique is a favourable combination for reducing zinc and lead contents in slags and return the metals to the material cycle. More zinc is won in flue dust bearing contents of lead than flue dusts from fuming plants, so that the flue dust can be used directly as a raw material in hydrometallurgical or pyrometallurgical zinc production.

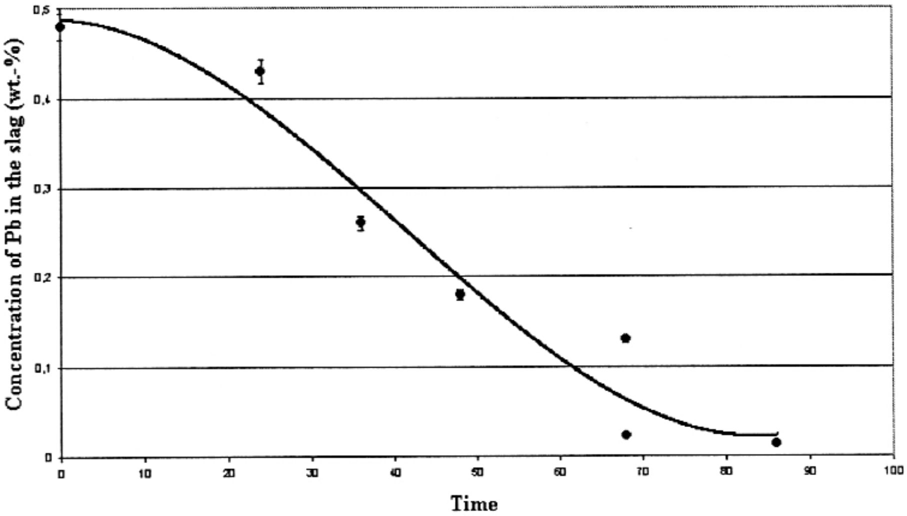

Figure 8. Lead content in IS-slag as a function of time

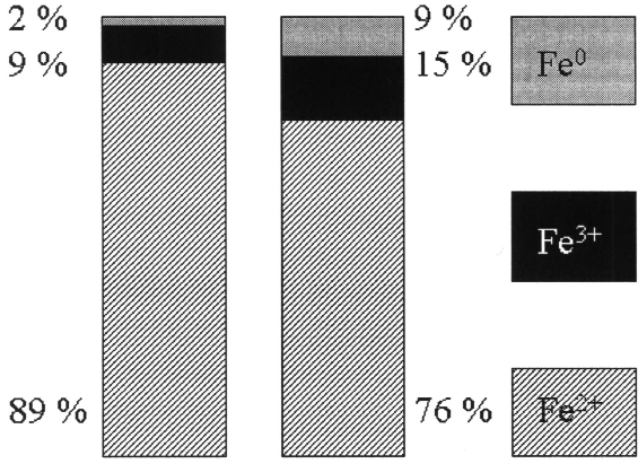

Figure 9. Distribution of relevant oxidation states of iron in the charged slag (left) and the final slag (right)

Lead is won as crude lead for refining. The treated slag is a useable raw material for building materials. The values of zinc and lead—which are critical for environmental friendly use—are reduced to concentrations below 1% of zinc and 0.1 % of lead.

The iron is mainly reduced to FeO and in parts to metallic iron (i.e. ~10% of total Fe) in this process. But under the prevailing conditions accretions are not expected.

Future work will concentrate on the influence of the sulphur content. The next test runs on a pilot scale are to be conducted early in 2004. Under what circumstances and to what extent zinc can be won as a metallic phase has yet to be modelled.

Acknowledgment

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